

i/p/15

09/914436

JC03 Rec'd PCT/PTO 27 AUG 2001

A process for producing compacted, free-flowing lacquer raw materials

The present invention relates to a process for producing compacted free-flowing nitrocellulose, which is moistened with water or alcohol, by pressing it through a plate provided with holes.

Nitrocellulose materials which have a low degree of esterification and a nitrogen content of up to 12.6 %, and which are mainly used in the lacquer industry, are produced by the esterification of cellulose materials with nitrating acids, which usually consist of a mixture of nitric acid, sulphuric acid and water.

After the nitrating acid has been removed, generally by intensive washing with water, and after the desired molecular weight of the nitrocellulose has been obtained by a thermal decomposition process, the nitrocellulose which is thus obtained, and which has a fibrous structure, has to be stabilised to prevent self-ignition. Various stabilising agents are used for this purpose.

Apart from the incorporation of plasticisers by admixture, the most commonly used method is the moistening of the nitrocellulose with alcohols and/or water. The nitrocellulose materials are usually sold with a moisture content of alcohols (such as ethanol, isopropanol or butanol for example) and/or water of 30 or 35 %, respectively. If the degree of moistening is less than 25 %, these partially esterified nitrocellulose materials have to be treated as "explosive substances" due to their increased hazard potential (Recommendations on the Transport of Dangerous Goods, 10th Edition, United Nations (1997)).

Due to their wool-like structure, fibrous nitrocellulose materials have apparent densities which range between 250 and 350 g/l on average. With regard to the shipping of products such as these, their low apparent density has a disadvantageous effect on packing and shipping costs. This is countered by tamping the fibrous nitrocellulose into the packaging container, such as a drum or carton for example.

"Express Mail" mailing label number ET146898643USDate of Deposit August 27, 2001

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Although the apparent density is in fact increased in this manner, the pourability of the nitrocellulose is at the same time reduced. This results in an increased labour requirement for emptying the nitrocellulose container.

5 A process for treating fibrous nitrocellulose materials is known, the object of which is to make them safer for transport and storage (GB-B-871 299). Compaction is effected by exerting a compressive force $P = 2M + 6400$ on the moist, fibrous nitrocellulose, where P is the force in pounds per square inch and M is the mean fibre length of the nitrocellulose in microns. The force, which preferably ranges between
10 15,000 and 17,000 psi (1110-1196 Kp/cm²), is applied by two rolls which are driven in counter-rotation without a separation.

The flake-like nitrocellulose which is thus obtained subsequently has to be broken into smaller pieces in a processing unit. Apart from its high capital and operating
15 costs, one considerable disadvantage of this process is the drying out of the nitrocellulose which occurs during roll compaction. In GB PS 871 299, all the compacted nitrocellulose materials which are obtained according to the examples have a moisture content less than 25 %, and are consequently explosive substances. It has been shown in practice that roll compaction under the action of high pressures
20 results in self-ignition phenomena which constitute a hazard for personnel and for the plant, particularly since there is always a very large amount of nitrocellulose in the roll gap.

A similar process principle is described in US PS 5 378 826. The aforementioned
25 disadvantages are also applicable there.

The object of the present invention was therefore to provide a new process for producing compacted, free-flowing nitrocellulose which prevents the nitrocellulose from drying out.

Sub
5 It has now surprisingly been found that compacted nitrocellulose can also be obtained by causing the circulating breakers (wheels) in a breaker mill, which breakers travel on a die (plate) which is provided with holes, to press the moistened nitrocellulose through the holes (e.g. bores) in the die (see Figure 1). The nitrocellulose lacquer raw material is thereby compacted. Underneath the die there is a shearing-off apparatus by means of which the granule-like preforms are brought to the desired length. The cross-sectional shape of the preforms is determined by the shape of the hole cross-section.

10 It should be emphasized that if defined process parameters are maintained there is hardly any drying-out of the nitrocellulose which is used, and thus the latter does not fall within the explosive substance range of less than 25 % moisture.

15 At least one breaker rotates in the breaker mill. Two breakers are customary, although more than 2 breakers can be provided. This depends on the size of the processing unit and on the diameter of the breakers. The compacted material which is obtained can vary from hard (with sharp edges) to soft (easily crushed with the fingers).

20 It is also possible, however, for the moistened nitrocellulose simply to be pressed through the die and thus to be compacted by a reciprocating movement of a wheel on a die which is provided with holes.

25 When the die contains circular holes, the pressing ratio P determines the consistency of the compacted nitrocellulose. P is defined as the ratio of the length of the bore to the diameter of the bore in the die:

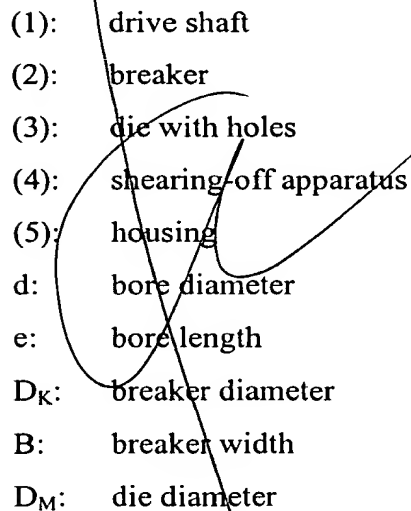
30
$$P = \frac{\text{length of bore}}{\text{diameter of bore}} .$$

The pressing ratio P should range between 0.5 – 5.0, preferably between 0.5 and 3.0.

It is also possible for the holes in the die to have a square, rectangular, oval or irregularly shaped cross-section. The number of holes per unit area of the die depends on the stability of the die. By means of compaction, it is possible to increase the apparent density of the nitrocellulose used by a multiple and thereby to obtain free-flowing material.

The uncompacted material is fed into the breaker mill via a metering device (such as a continuous screw or belt for example). It is also possible to provide the breaker mill with an inert atmosphere, e.g. nitrogen or carbon dioxide. The following examples describe the process in greater detail, but do not limit it.

Figure 1:

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- (1): drive shaft
 - (2): breaker
 - (3): die with holes
 - (4): shearing-off apparatus
 - (5): housing
 - d: bore diameter
 - e: bore length
 - D_K : breaker diameter
 - B: breaker width
 - D_M : die diameter

Example 1

Nitrocellulose corresponding to Standard A 30, moistened with 34.6 % ethanol, was continuously fed at 210 kg/h into the breaker mill (die diameter: $D_M = 175$ mm, breaker diameter: $D_K = 130$ mm, breaker width: $B = 27$ mm, number of breakers: 2, drive shaft speed = 150 rpm). The pressing ratio was 2 (hole diameter: $d = 6$ mm, hole shape: circular, length of bore: $l = 12$ mm). The distance from the shearing apparatus to the underside of the die was 20 mm. The compacted material had a moisture content of 34.2 % and an apparent density of 578 g/l. The preforms remained free-flowing in a 100 litre drum even after being stored therein for eight weeks.

Examples 2 – 7

These examples were carried out as described for Example 1. The test parameters are given in Table 1.

Example	NC Standard	Moistening agent	Moisture content		Throughput	Apparent density		Die			Drive shaft speed (rpm)
			before (%)	after (%)		before (g/l)	after (g/l)	D (mm)	L (mm)	P	
1	A 30	ethanol	34.6	34.2	210	383	578	6	12	2	150
2	E 27	ethanol	32.4	31.5	116	253	565	6	12	2	150
3	E 22	ethanol	32.0	31.7	130	196	556	10	20	2	150
4	E 34	ethanol	34.1	31.9	332	438	602	10	20	2	150
5	E 24	isopropanol	35.2	35.0	190	191	556	8	16	2	150
6	A 27	ethanol	30.9	30.4	133	380	539	6	6	1	150
7	E 22	ethanol	32.8	31.7	57	196	526	6	6	1	201